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Early Precambrian tectonothermal events of the North China Craton: Constraints from *in situ* detrital zircon U-Pb, Hf and O isotopic compositions in Tietonggou Formation

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Clastic sediments and sedimentary rocks are widely used for understanding the formation and evolution of the continental crust. The Tietonggou Formation outcrops in the Xiaoqinling region at the southern margin of the North China Craton (NCC) and has unconformable contacts with the underlying Taihua Complex and overlying Paleoproterozoic Xiong'er Group. It mainly consists of quartzite and its protoliths are mature terrigenous clastic rocks. On the basis of the ages of the youngest detrital zircons from the quartzites and the ages of the Xiong'er Group, the depositional ages of the protoliths of the Tietonggou Formation were well constrained to 1.91–1.80 Ga. The U-Pb isotopic data of detrital zircons from the Formation show a major age peak at ~2.1 Ga, which is consistent with 2.2–2.0 Ga magmatism in the Trans-North China Orogen of the NCC. Taking into account the texctural and compositional maturity of the Tietonggou Formation quartzite, the ~2.1 Ga lithologic units in the Trans-North China Orogen are interpreted as the major source of the Tietonggou Formation. The majority of these ~2.1 Ga detrital zircons mostly have high δ^{18}O values (>6.5‰) and negative $\varepsilon_{\mathrm{Hf}}(t)$ values (–7.8–0.0), with corresponding Hf model ages significantly older than their crystallization ages, indicating that these zircons formed from the partial melting of ancient continental crust. The majority of the 2.8–2.7 Ga and ~2.5 Ga detrital zircons from the Tietonggou Formation had positive $\varepsilon_{\mathrm{Hf}}(t)$ values, and mantle-like δ^{18}O values, suggesting that the NCC has experienced two stages of significant crustal growth in the Neoarchaean at 2.7 and 2.5 Ga, respectively. The Hf isotopic data of detrital zircons from Paleoproterozoic metasedimentary rocks in the Trans-North China Orogen varied mainly toward the reduction of the radiogenic Hf isotope and gradually show a similar trend of the isotope trajectories of crustal evolution. This reveals that the NCC probably has not developed a long-lived subduction to complete the final assembly of the NCC. Alternatively, these maybe imply that the tectonic setting of the NCC substantially changed at ~2.1 Ga, the reduction of the radiogenic Hf isotope could be attributed to the rollback of the subducting slab.

North China Craton, Tietonggou Formation, detrital zircon, Hf isotope, O isotope

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Clastic sediments and sedimentary rocks are representative samples for studying the formation and evolution of a craton [1,2]. In contrast to magmatic rocks, detrital minerals, particularly detrital zircons from sedimentary rocks, are resistant to mechanical abrasion and chemical weathering, and thus can record valuable information of source rocks that no longer exist. Furthermore, individual zircons can be analyzed for U-Pb isotopes to identify a specific sequence of major magmatic events, and their Lu-Hf and O isotopic compositions can provide information on continental crustal evolution. Detrital zircons therefore have offered useful information of the provenance of clastic sediments and sedimentary rocks, regional magmatic events and the evolution of the continental crust, such that they have recently become one of the hotspots in geological research [1–4]. This study investigated U-Pb geochronology and Hf-O iso-

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topic compositions of detrital zircons from the Paleoproterozoic Tietonggou Formation quartzite at the southern margin of the North China Craton (NCC) to constrain the compositions and possible sources of the sediments. In addition, a comprehensive regional study examined the early Precambrian tectonothermal events to provide insights into the early Precambrian crustal formation and evolution of the NCC.

1 Geological setting and sample collection

Over the past decade, it has been broad consensus that the continental nucleus of the NCC formed at the Paleoarchean, followed by amalgamation of a number of mico-continental blocks. However, controversies still exist regarding the number of continental block, and when and how assembled to form the NCC. There are two different models to explain time of final amalgamation of NCC. Some researchers believed that the NCC formed by assembling of several micro-continental blocks at ~2.50 Ga [5–8], whereas others proposed that the final collision of NCC occurred at ~1.85 Ga [9,10]. Conventional, the final amalgamation and cratonization of the NCC was considered to be related to the Lüliang movement at the late Paleoproterozoic (~1.8 Ga) [11], which are characterized by unconformity between the Paleo-Mesoproterozoic sedimentary strata and metamorphosed Archean basement in different regions. For example, the Paleoproterozoic Yejishan, Hutuo, Zhongtiao, Songshan Groups, and Tietonggou Formation were deposited overlies the Neoarchean–Paleoproterozoic basement in the Lüliang, Wutai, Zhongtiao, Songshan, Xiaoqinling regions in the Trans-North China Orogen, respectively.

In the Henan-Shaanxi province on the southern NCC, the Neoarchean-Paleoproterozoic rocks extend in an E-W direction and consist of the Taihua and Dengfeng Complexes, the Angou, Xiong'er and Songshan Groups and the Tietonggou Formation. Of these, the lower subgroup of the original Taihua Group [12,13] (which disintegrated from the Taihua Complex), the Dengfeng Complex [14,15] and the Angou Group [16] developed in Neoarchean, whereas the upper subgroup of the Taihua Group [12], the Songshan [17,18], Xiong'er Group and the Tietonggou Formation were formed in Paleoproterozoic [19].

The Tietonggou Formation outcrops in the Xiaoqinling region. In 1958, the Bureau of geology and mineral resources of Shaanxi Province (BGMRSP) has established a typical geologic section for the Tietonggou Formation in Bayuan area, Lantian County, Shaanxi Province [20]. The formation consists of a succession of clastic sendiematry rocks 315–2850 m in thickness and unconformably overlies the Taihua Complex (Figure 1). In the Bayuan region, the Tietonggou Formation was unconformably overlain by the Mesoproterozoic Xiong'er Group. This unconformity present an orogeny, named the "Luanchuan movement" by some Chinese geologists, which occurred coevally with the "Lüliang movement" and "Zhongtiao movement" in Lüliang and Zhongtiao area of Shanxi Province, respectively. The Tietonggou Formation is composed mainly of quartzite, pebbly quartzite, and muscovite quartzite (Figure 2). Its protoliths were mature terrigenous clastic rocks. The ripple marks and cross-bedding structures still preserved in the quartzite, which was considered to have deposited in littoral sedimentary environment [20]. The Tietonggou Formation differs from the underlying Taihua Complex and the overlying Xiong'er Group in rock assemblages and metamorphic grade. The Taihua Complex consists of TTG gneisses, biotite (hornblende) plagiogneiss and amphibolite interbedded with (magnetic) quartzite, graphite gneiss, leucoleptite and marble, which have undergone amphibolite-granulite facies metamorphism. The overlying Xiong'er Group is composed of volcanic rocks (including basaltic andesites, andesites, rhyolitic lavas and minor pyroclastic rocks) with low grade of metamorphism up to greenschist-facies [21,22].

In this study, two quartzite samples, 01TT–01 and 01TT–02, were collected from the Tietonggou Formation in Bayuan area, Lantian County (Figure 2). The rocks are mostly white or gray in color and display a massive structure. They occur as medium-coarse grains with high psephicity and consist of over 90% quartz. Accessory minerals are zircon and monazite. Quartzs in quartzites are commonly xenomorphic granular with undulose extinctions and often show dentate contact between quartz grains. Under the microscope, 01TT–01 has fine grained with >90% quartz and 1%–2% rock debris and 01TT–02 has coarse quartz with more feldspar (~5%).

2 Analytical methods

Zircons were extracted by using a combined technique of heavy liquid and magnetic separation, and then handpicked under a binocular microscope, mounted in epoxy resin and polished until the grain centers were exposed. To reveal their internal structures, cathodoluminesce (CL) imaging was undertaken using a Quanta 400FEG environmental scanning electron microscope equipped with an Oxford energy dispersive spectroscopy system and a Gatan CL3+ detector.

Zircon U-Pb-Hf isotopes analyses were performed at the State Key Laboratory of Continental Dynamics, Northwest University, Xi'an, China. The U-Pb dating was conducted on an Agilent 7500a ICP–MS instrument equipped with a 193 nm ArF-excimer laser and a homogenizing, imaging optical system. Fixed spot size of 20–30 μm with a laser repetition rate of 5 Hz was adopted throughout this study. Helium was used as carrier gas to provide efficient aerosol delivered to the torch. The standard silicate glass NIST 610 was used to optimize the machine to obtain a maximum signal intensity (^{238}U signal intensity >460 cps/ppm) and

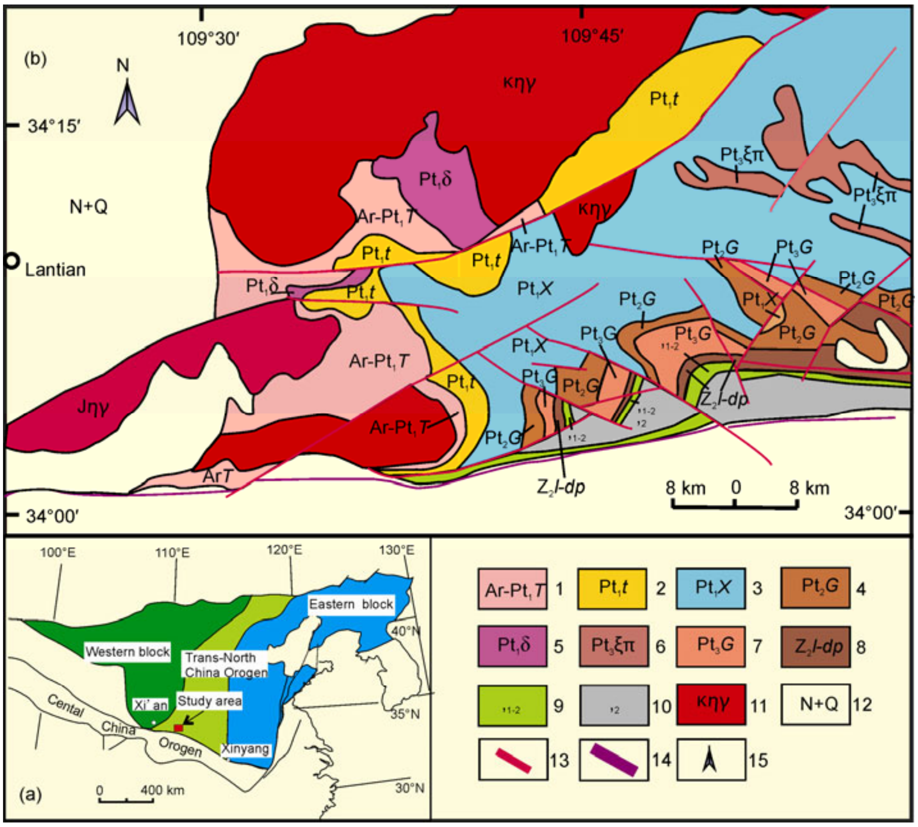

Figure 1 Geological sketch map of Lantian area (modified after [20]). 1, Neoarchean–Paleoproterozoic Taihua Complex; 2, Paleoproterozoic Tietonggou Formation; 3, Paleoproterozoic Xiong'er Group; 4, Mesoproterozoic Guandaokou Group; 5, Paleoproterozoic diorite; 6, Neoproterozoic orthophyre; 7, Neoproterozoic Gaoshanhe Group; 8, Sinian strata; 9, Early–middle Cambrian strata; 10, Middle Cambrian strata; 11, granite; 12, Tertiary and Quaternary; 13, Heigou fault; 14, fault; 15, north.

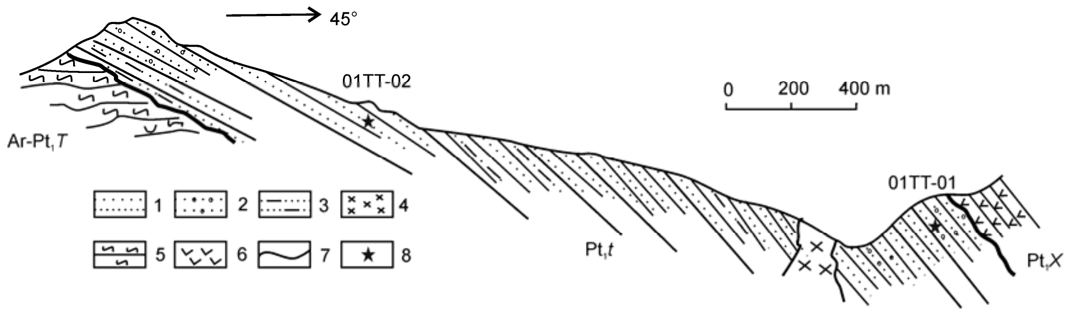

Figure 2 The geological section of Tietonggou Formation in Liantian County (modified after [20]). 1, Quartzite; 2, pebbly quartzite 3, mica-quartz schist; 4, granite; 5, Neoarchean–Paleoproterozoic Taihua Complex; 6, Paleoproterozoic Xiong'er Group; 7, unconformable contact; 8, sampling site.

low oxide production (ThO/Th<1%). The laser ablation strategy is a single spot in one zircon. The ICP-MS measurements was carried out using time resolved analysis operating in fast peak jumping mode and DUAL detector mode using a short integration time. ^{207}Pb/^{206}Pb, ^{206}Pb/^{238}U, ^{205}Pb/^{235}U and ^{208}Pb/^{232}Th ratios were calculated using the GLITTER 4.0 program (Macquarie University), and then corrected using the Harvard zircon 91500 as external standard with a recommended ^{206}Pb/^{238}U age of 1065.4±0.6 Ma to corrected for both instrumental mass bias and depth-dependent elemental and isotopic fractionation. U, Th and Pb concentrations were calibrated by using ^{29}Si as an internal standard and NIST SRM 610 as an external standard. Concordia diagrams and weighted mean calculations were made using Isoplot (ver 2.49) [23].

Zircon Hf isotopic analyses were undertaken on a Nu Plasma HR MC-ICP-MS (Nu Instruments Ltd., UK) equipped with a GeoLas 2005 193 nm ArF-excimer laser-ablation system. Analyses were carried out using spot size of 44 μm and He was also used as carrier gas. The laser repetition rate is 10 Hz and the energy density applied is 15–20 J/cm^{-2}. Raw count rates for ^{172}Yb, ^{173}Yb, ^{175}Lu,

^{176}(Hf + Yb + Lu), ^{177}Hf, ^{178}Hf, ^{179}Hf and ^{180}Hf were collected simultaneously. The isobaric interference of ^{176}Lu on ^{176}Hf was corrected by measuring the intensity of an interference-free ^{175}Lu isotope and also a recommended ^{176}Lu/^{175}Lu ratio of 0.02669 to calculate ^{176}Lu. Similarly, the interference of ^{176}Yb on ^{176}Hf was corrected by measuring an interference-free ^{172}Yb isotope and using a ^{176}Lu/^{172}Yb ratio of 0.5886 to calculate ^{176}Hf/^{177}Hf ratios [24]. Time-dependent drifts of Lu-Hf isotopic ratios were corrected using a linear interpolation according to the variations of 91500 and GJ–1.

A decay constant for ^{176}Lu of 1.867×10^{-11} a^{-1} [25], the present day chondritic ratios of ^{176}Hf/^{177}Hf = 0.282772 and ^{176}Lu/^{177}Hf = 0.0332 [26] were adopted to calculate $\varepsilon_{\mathrm{Hf}}$ values. Single-stage mantle model ages (T_{DM1}) were calculated by reference to depleted mantle with a present-day ^{176}Hf/^{177}Hf ratio of 0.28325, similar to that of average MORB and ^{176}Lu/^{177}Hf ratio of 0.0384 [27]. The two-stage model age (T_{DM2}) was calculated by projecting the initial ^{176}Hf/^{177}Hf of zircon back to the depleted mantle growth curve using ^{176}Lu/^{177}Hf = 0.015 for the average continental crust [28].

Zircon oxygen isotopes were measured using the Cameca IMS 1280 ion microprobe at the Institute of Geology and Geophysics, Chinese Academy of Sciences in Beijing. The spot size was ~20 μm in diameter. The TEMORA 2 zircon standard during the course of this study yielded a weighted mean δ^{18}O = 8.14 ± 0.20‰ (n=13, 2σ), which is consistent with the reported value. Analytical procedures were similar to those described in reference [29].

3 Results

3.1 Zircon U-Pb ages

Over one thousand zircon grains were separated from samples 01TT–01 and 01TT–02. Zircon crystals are mostly translucent, light brown in color, subhedral to euhedral in morphology. They varied in size (up to 200 μm) and highly round, indicating their strong erosion and long distance of transportation. Most of zircon grains developed intense oscillatory zoning (Figure 3), suggesting a magmatic origin. According to the rules defined by Anderson [30], for provenance studies, the application of detrital zircon data may be a combination of random and nonrandom selection for analysis. The random analysis should comprise 35–70 zircon grains or more, depending on the complexity of the age distribution; and then take non-random analysis on certain dated zircons for trace element and Lu-Hf isotope data to acquire additional, valuable information. In this study, more than 70 zircon grains have been analyzed for each sample to satisfy the statistical requirement. In addition, old zircon (>1000 Ma) grains usually underwent Pb-loss in certain time after they formed. ^{207}Pb/^{206}Pb ratios may remain constant at the same original condition and similar geology environment, such that ^{207}Pb/^{206}Pb ages were not disturbed and may represent the real crystallization age of rocks. If a zircon U-Pb age is concordant, the ^{207}Pb/^{206}Pb and ^{206}Pb/^{238}U ages would be the same, so the data with more than 10% discordance are completely rejected by comparison of ^{206}Pb/^{238}U and ^{207}Pb/^{206}Pb ages.

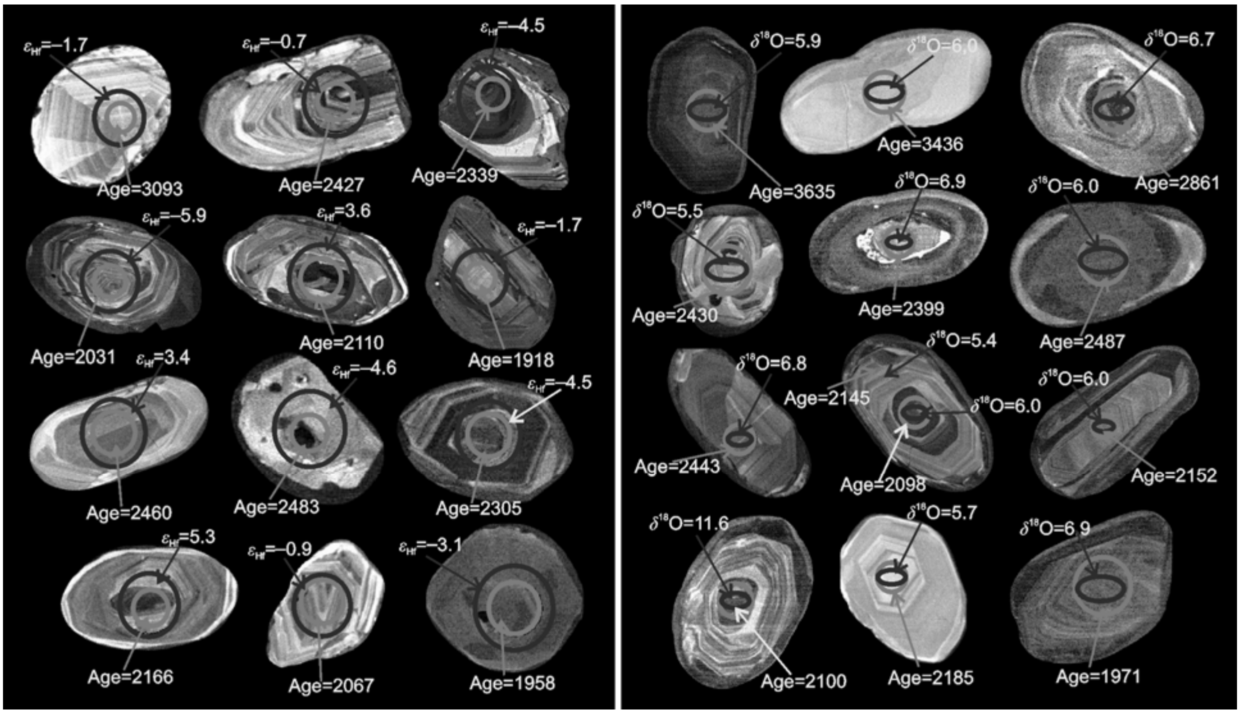

Figure 3 Cathodoluminescence (CL) images of typical zircon grains from the Tietonggou Formation.

A total of 115 zircon U-Pb isotopic analyses were obtained from sample 01TT–01 (Table S1). Results showed that the concentration of common lead in all of the zircons is lower than the limit of detection of ICP-MS (0.039 ppm), except for a few grains contaminated 0.1% common lead in zircons. The majority of analysis spots are distributed on or near the Concordia line, only a few are discordant (Figure 4). The $^{207}Pb/^{206}Pb$ ages range from 1918 to 3483 Ma. As the probability diagram of zircon $^{207}Pb/^{206}Pb$ ages for sample 01TT–01 show that there is a prominent age peaks at 2080 Ma (Figure 4), which constitutes about 80 percent of the total data.

In order to obtain multiple isotopes results, Sample 01TT–02 was divided into subsamples 01TT02–2010 and 01TT02–2011. One hundred and ten zircon grains were dated by the LA-ICPMS for subsample 01TT02–2010, it is worthy to note that the oldest zircon grain gives a $^{207}Pb/^{206}Pb$ age of 3635 Ma, the youngest zircon dated has a $^{207}Pb/^{206}Pb$ age of 1952 Ma, the latter could be taken as the maximum depositional age for this rock. The probability diagram of zircon $^{207}Pb/^{206}Pb$ ages for the sample displays a primary peak at 2100 Ma that account for about 40% of the analyzed data. Moreover, two minor peaks at 2720 Ma and 2476 Ma also can be recognized.

Following zircon oxygen isotopes analysis, 126 U-Pb analyses were obtained for the subsamples 01TT02–2011, the $^{207}Pb/^{206}Pb$ ages range from 1913 to 3436 Ma with a primary peak at 2100 Ma and a minor peak at 2450 Ma

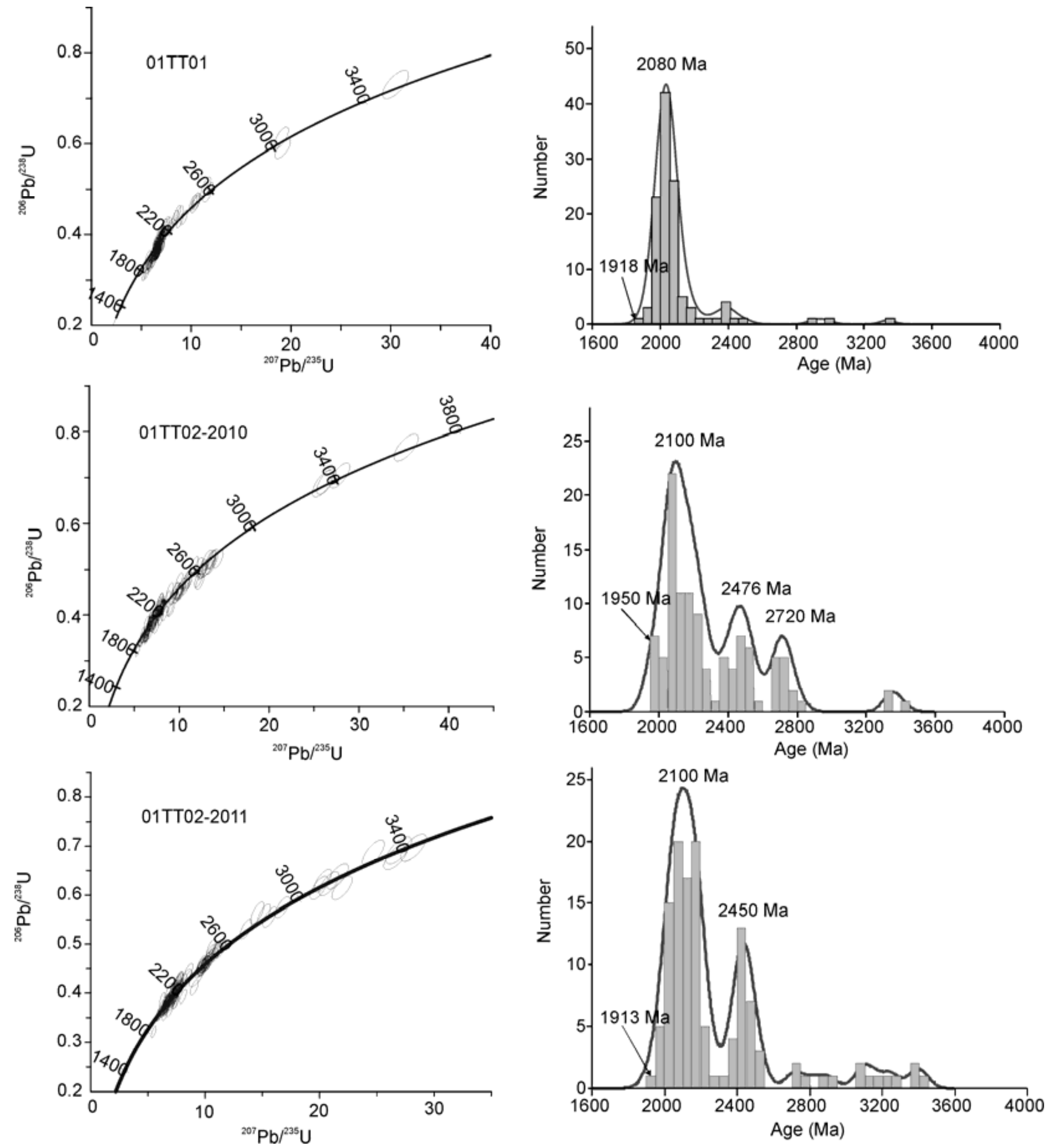

Figure 4 Left panels show U-Pb concordia plots of concordant detrital zircons from the Tietonggou Formation. Right panels show corresponding relative probability plots of $^{206}/^{207}Pb$ ages for concordant detrital zircons.

(Figure 4).

3.2 Zircon Hf-O isotope compositions

A total of 225 detrital zircons from sample 01TT–01 and 01TT02–2010 were analyzed for Hf isotope and the results are available in Table S2. ^{176}Lu/^{177}Hf ratios of all zircons are less than 0.002 with an average of 0.0007, indicating a low radiogenetic growth of ^{176}Hf. The $\varepsilon_{\rm Hf}(t)$ values were calculated by their corresponding ^{207}Pb/ ^{206}Pb ages. As shown in Figure 5(a), all the detrital zircons older than ~2.5 Ga exhibit negative $\varepsilon_{\rm Hf}(t)$ values. The majority of ~2.5 Ga zircon grains have positive $\varepsilon_{\rm Hf}(t)$ values, up to 6.7. Whereas ~2.1 Ga zircons mostly have variable negative $\varepsilon_{\rm Hf}(t)$ values, down to –20. The two stage Hf continental model ages of detrital zircons cluster between 2500 and 2700 Ma. Three old detrital zircons with ^{207}Pb/^{206}Pb ages of 3344, 3401 and 3635 Ma have $T_{\rm DM2}$ ages of 4040, 4326, and 4373 Ma, respectively.

One hundred and twenty-six zircon grains from sample TT02–2011 were selected for O isotope analyses (Table S3). As shown in Figure 6, the majority measured zircons which older than ~2.5Ga have mantle-like oxygen isotopes (5.0‰–6.5‰). Some δ^{18}O values of ~2.5 Ga zircons have obviously higher than those of zircons in equilibrium with mantle magmas (up to 8.7‰). By comparison, the ~2.1 Ga zircons show significantly high δ^{18}O values (up to 11.6‰) which obviously higher than those of mantle-derived zircons.

The δ^{18}O compositions of 2.8–2.7 Ga zircons (black square) are cited from reference [34]. The shaded field depicts the δ^{18}O value (5.3±0.6‰, 2SD) of the mantle-derived zircons, and the δ^{18}O value of 6.5‰ is the upper limit of the zircons from primitive magmas considering the analytical uncertainty of SIMS.

4 Discussion and conclusions

4.1 Deposition time of the Tietonggou Formation

Although the Tietonggou Formation is conventionally considered to have formed in Paleoproterozoic [20], the depositional age of the formation has no been well constrained. Generally speaking, the youngest detrital zircon obtained from a low-grade metsedimentary rocks can be used to determine its maximum depositional time, with the proviso of no disturbance in the U-Pb isotopic system of aged detrital zircon. This approach is most widely applied to Precambrian successions where biostratigraphy cannot be used. In this study, the youngest concordant detrital zircons obtained from each quartzite samples of Tietonggou Formation was 1918 ± 58 Ma (01TT01–97) and 1913 ± 53 Ma (01TT02–2011–136), respectively. Therefore, the age of 1913 Ma can be regarded as the maximum depositional age of the Tietonggou Formation. However, sometime the youngest age of detrital zircon obviously far older than the depositional age of metsedimentary rocks and might has no geological significance. For example, Diwu et al. [35] have dated 84 detrital zircons from the Changlongshan Formation of the Neoproterozoic Qingbaikou sedimentary sequence in Qinhuangdao city, Hebei province. The youngest detrital zircon obtained is 2487 Ma, which is far older (>1.5 Ga) than the depositional age of the metasedimentary protoliths. Therefore, the minimum depositional age of the Tietonggou Formation should be also constrained.

As mentioned above, the Xiong'er Group unconformably overlies the Paleoproterozoic Tietonggou Formation. Thus, the minimum depositional age of the Tietonggou Formation can be constrained by the formation age of the Xiong'er Group. In the last three decades, there has been considerable debate over the petrogenesis and tectonic settings of the

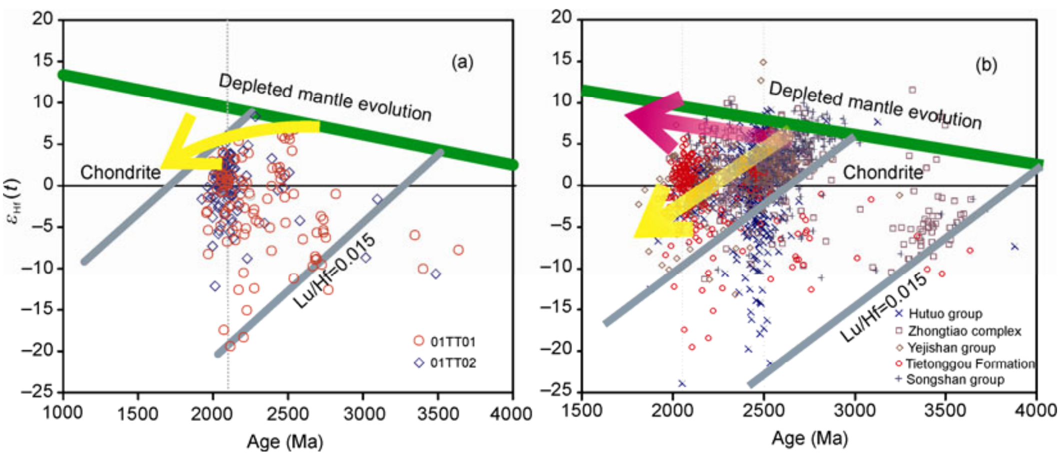

Figure 5 Relationship between $\varepsilon_{\rm Hf}(t)$ values and U-Pb ages for zircons from the Tietonggou Formation (a) and the detrital zircons of the Paleoproterozoic metasedimenatry from the Trans-North China Orogen. Data sources: Huotuo group [31], Zhontiao complex [32], Yejishan group [33], Songshan group [18]. Note that $\varepsilon_{\rm Hf}(t)$ values of zircons of the Paleoproterozoic metasedimenatry from the Trans-North China Orogen show a the progression towards decreasingly juvenile Hf isotope compositions during the period of 2.5–1.8 Ga. In contrast to the yellow array in (a) and (b), the pink array in (b) shows a progression towards increasingly juvenile Hf isotope compositions after Neoarchean.

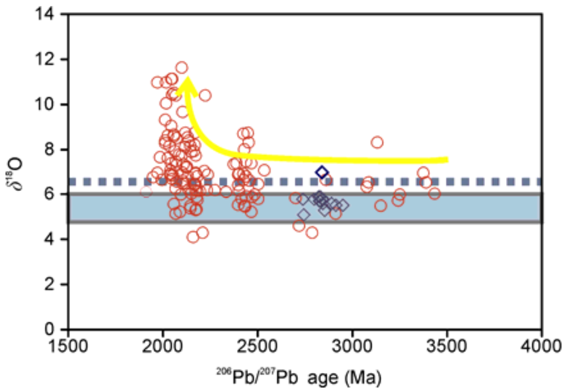

Figure 6 $\delta^{18}O$ compositions of dated detrital zircons of Tietonggou Formation (circles).

Xiong'er volcanic rocks, however available SHRIMP and LA-ICPMS U-Pb zircon age data indicate that the Xiong'er Group formed at 1.80–1.75 Ga [19,21–22]. Although minor ~1.45 Ga felsic volcanic rocks are reported in Xiong'er Group, which possibly represent a later magmatic activity in the southern NCC [22]. Because Cui et al. [36] recently identified some intermediate-acid intrusive rocks that invaded the uppermost Majiahe Formation of the Xiong'er Group, SIMS zircons and baddeleyites U-Pb analyses indicate that those rocks were emplaced at 1778–1789 Ma, therefore it constrains the minimum age limit of Xiong'er Group volcanic rocks. Taken together, the depositional age of the Tietonggou Formation can be constrained at 1.91–1.80 Ga.

4.2 Provenance of the Tietonggou Formation

More recently, Wan et al. [2] compiled a total of 7586 U-Pb ages of zircons from the early Cambrian basement rocks of the NCC, the result show a notable U-Pb age peak at ~2.5 Ga in the period of Archean, which are characteristic of the NCC and distinctly different from most other cratons worldwide, where ~2.7 Ga age are predominant. Whereas the detrital zircon ages from the two quartzite samples of Tietonggou Formation show broadly similar age patterns with one major age population of ~2.1 Ga (Figure 4). In addition, a significant number of ~2.5 Ga detrital zircon occurs as a minor age population in sample 01TT–02 than in 01TT–01. In order to compare potential provenance similarities of two samples, Kolmogorov-Smirnov (K-S) test can be provides a statistical basis for hypothesis testing. The K-S test determines a probability (P), if P-value >0.05, it is means that there is 95% confidence that the age cumulative probability plots reflect that the two detrital zircons samples come from indistinguishable sources. The age cumulative probability plots of the two quartzite samples (01TT–02 and 01TT–01) show a systematic difference and give P-value < 0.01 by the K-S test, which imply that detrital zircons in the two quartzite samples were possibly derived from significantly different sources.

Detrital zircons form the two quartzite samples in the Tetonggou formation exhibit a predominant age population of ~2.1 Ga (Figure 4). This age is well consistent with the ages of the lithologic units in the Trans-North China Orogen. A large number of such aged Paleoproterozoic intrusive rocks are reported, including Changchengling granite (2113 ± 8 Ma) [37] and ~2.1 Ga potassic granite [38] in the Hengshan area; Wangjiahui granite (2117 ± 18 Ma) [39] and Dawaliang granite (2176 ± 12 Ma) [40] in the Wutai region; Nanying gneiss (2077 ± 13 Ma) [41] and Gangnan gneiss (2045 ± 64 Ma) [42] in the Fuping region; Xuting granite (2090 ± 10 Ma) in the Lüliang-Zanhuang region [43]; tonalite (2199 ± 11 Ma), granodiorite (2180 ± 7 Ma), and monzogranite (2173 ± 7 Ma) in the Chijianling–Guandishan region [44]; and feldspar porphyry (2189 ± 6 Ma) in the Dujiagou region [45]. In addition, contemporaneous volcanic rocks were also found in the Trans-North China Orogen of NCC. For example, metavolcanic rocks and metarhyolites in the Yejishan Group and the Lüliang Group are yielded zircon ages of 2.3–2.0 Ga [46–48]. Du et al. [49] reported a SHRIMP zircon agr of 2140 ± 14 Ma for basaltic andesite from the lower part of the Hutuo Group. Peng et al. [50] recognized 2147 Ma meta-mafic dykes in the Trans-North China Orogen of NCC. Wang et al. [51] found 2193 Ma gabbro in the Hengshan region. Furthmore, 2.1 Ga volcanic rocks were reported in the Zhongtiao complex [52]; gneissic granites in the Lushan region gave a U-Pb age of 2139 Ma [53] in the southern margin of the NCC.

The protoliths of the Tietonggou Formation are considered to be a set of terrigenous clastic rocks and mainly consist of quartzes. In general, quartzes in intermediate-acid volcanic rocks occur as single crystals and are transparent without undulose extinction. Whereas, quartz grains in sedimentary rocks mostly occur as monocrystalline without wavy extinctions and it can be observed that the quartz overgrowth crystallized from original siliceous surrounds around the rim of the original quartz grain. Microscopic observation show that quartz grains from the Tietonggou Formation quartzite do not have typical quartz grains in either volcanic or sedimentary rocks as mentioned above. In fact, these quartz grains mostly have suture-like contact boundaries or apparent undulose extinction, indicating that they were derived from metagranitic rocks. The high compositional maturity of the quartz sandstone suggests the source materials have experienced long-distance transportation. Taking into account the high textural and compositional maturity of Tietonggou Formation quartzite, its major sources are considered to be derived from the ~2.1 Ga lithologic units in the Trans-North China Orogen.

4.3 Implications for early Precambrian crustal evolution in the NCC

In recent years, available SHRIMP and LA-MC-ICPMS U-Pb zircon ages and Hf isotopic compositions of the early

Precambrian rocks have revealed that the NCC experienced two episodes crustal growth in the Neoarchean at ~2.7 Ga and ~2.5 Ga, respectively [54]. The ~2.7 Ga rocks are locally exposed in the Lushan county of Henan Province [12,13,34], the Zhongtiao Mountains of Shanxi Province, western Shandong Province [55,56] and eastern Jiaodong peninsula [55]. This is consistent with the global juvenile crustal growth event at ~2.7 Ga, crustal formation of orogenic belts and supercontinent cycles [57]. In compare with the ~2.7 Ga rocks, ~2.5 Ga rocks are widespread in the NCC, Large volumes of TTG gneiss, mantle-derived granites and minor supracrustal rocks were formed during this time, therefore it is proposed that ~2.50 Ga is the most important period of tectonothermal events and crustal growth in the NCC. However some researchers believe that the widespread ~2.5 Ga rocks are related to reworking of the crustal rocks [58].

The value of initial epsilon (hafnium ($\varepsilon_{\mathrm{Hf}}(t)$)) of zircon insights provided information about the crustal growth and evolution. If $\varepsilon_{\mathrm{Hf}}(t)$ is negative, the zircon of age t formed from protoliths in the continental crust. If $\varepsilon_{\mathrm{Hf}}(t)$ is positive, these reveal information about the growth of the continental crust by addition of volcanic or plutonic rocks derived from the mantle. However, Hf isotopes of zircons difficult distinguish whether the magmas may contain mixed contributions from both juvenile and recycled (metasedimentary) sources, or an ancient mafic underplate [59]. The oxygen isotope ratio (δ^{18}O) of zircon has been shown to effectively trace the contribution of supracrustal material to magma. The zircon δ^{18}O is generally preserved through subsequent high-temperature hydrothermal and high grade metamorphism. Zircons in equilibrium with pristine mantle-derived melts theoretically have δ^{18}O values of 5.3 ± 0.3‰. However, zircons crystallized from a mixed source magma which involves a sedimentary cycle or hydrothermal alteration have elevated δ^{18}O values [60,61]. Thus, combination of Hf and O isotopes can be used to distinguish zircons derived from a mantle sources or a crustal source region with a high sedimentary component [4,62].

Despite zircon oxygen isotope data from Archaean rocks of the NCC is limited, as the available data show that the majority of 2.8–2.7 Ga [34] and ~2.5 Ga (detrital) zircons have mantle-like δ^{18}O values in the range of 5.0‰–6.5‰ (Figure 6), and high positive $\varepsilon_{\mathrm{Hf}}(t)$ values (Figure 5), suggesting that the NCC has experienced two stages of significant crustal growth in the Neoarchaean at 2.7 and 2.5 Ga, respectively. A few ~2.5 Ga detrital zircons have high δ^{18}O values (up to 8.7‰), which above the upper limit of the zircons from primitive magmas considering the analytical uncertainty of SIMS (6.3‰), indicating that ~2.5 Ga tectono-thermal event represents an important period of crustal growth with minor ancient crust reworked in the North China Craton. As shown in Figure 4, 85% of the zircons in the Tietonggou Formation quartzite give ages of ~2.1 Ga. They have negative $\varepsilon_{\mathrm{Hf}}(t)$ values (Figure 5(a)), ranging from –7.8 to 0.0, and their two-stage Hf model ages obviously older than corresponding crystallization ages, suggesting that ~2.1 Ga zircons may have come from recycled ancient crust with a relatively long crustal residence time. As we noticed that some ~2.1 Ga detrital zircons have positive $\varepsilon_{\mathrm{Hf}}(t)$ values, however its $\varepsilon_{\mathrm{Hf}}(t)$ values significantly lower than contemporary depleted mantle. In addition, these detrital zircons have high δ^{18}O values (>6.5‰) (Figure 6), further indicating that the ~2.1 Ga magmatic event in the NCC was dominated by the recycling of crustal materials with minor mantle materials involved.

4.4 Early Precambrian tectonic evolution of the NCC

The early Precambrian tectonic evolution of the NCC is still controversial. Zhai et al. [5,6] suggested that the NCC divided into six blocks and they were joined into the NCC at ~2.5 Ga, following by Paleoproterozoic rifting-subduction-accretion-collision tectonics and subsequent high-grade granulite facies metamorphism-granitoid magmatism during ca. 2.0–1.82 [63]. Zhao et al. [9,10] divided the NCC into the Eastern and Western Blocks and the Trans-North China Orogen. They suggested that the Western Block was formed by the collision of the Ordos Block and the Yinshan Block along the Khondalite Belt at ~1.9 Ga. The Western Block continually subducted eastward from the end of the Archean. Finally, the Western and Eastern Blocks collided along the Trans-North China Orogen to form the coherent basement of the NCC at ~1.85 Ga. Kusky et al. [7,8] also provided a model to divide the NCC into the Eastern Block, Western Block, and Central Orogenic Belt which underwent arc/continent collision at 2.5 Ga. Recently, Faure and Trap [64,65] proposed the Fuping Block lies between the Eastern and Western Blocks of the NCC. These three blocks were separated by the Lüliang and Taihang Oceans. At ~2100 Ma, the Taihang Oceans closed by westward subducttion beneath the Fuping Block, leading to the formation of the Wutai arc, Lüliang back-arc basin and the 2100 Ma orogenic belt. Thereafter, the Fuping Block and the Western Block collided to form the NCC during 1900–1800 Ma [9,10].

As mentioned above, clastic sediments and sedimentary rocks are widely used for understanding the formation and evolution of the continental crust [1,2]. To unravel the Neoarchean–Paleoproterozoic tectonic evolution of the NCC, this study summarized available detrital zircon Hf isotope data of Paleoproterozoic metasedimentary rocks from the Tietonggou Formation as well as those from the Hutuo, Yejishan and Songshan Groups, and Zhongtiao Complex in the Trans-North China Orogen of the NCC. Figure 5(b) shows that the Neoarchean (2.7–2.5 Ga) was an important period of crustal growth in the NCC. During this period, a large amount of mantle materials were added to form the juvenile crust, as deciphered by from the highest positive $\varepsilon_{\mathrm{Hf}}(t)$ values that are not only in agreement with the initial Hf isotope ratios of coeval depleted mantle but also in association with the youngest Hf model ages identical to the timing of zircon growth from mantle-derived magmas. The Neoarchean rocks consist of tonalite-trondhjemite-granodiorite gneisses, the syn- or post-potassic granite and supercrustal rocks. ~2.5 Ga magmatism has occurred with the contemporaneously metamorphism, which has been interpreted as that crustal growth in the Neoarchean was related to a mantle plume [67]. Afterwards, the NCC behaved as a stable continent block during the early Paleoproterozoic (2500–2350 Ma). The available O-Hf isotopic data of detrital from metasedimentary rocks in the Trans-North China Orogen indicate that the ~2.1 Ga is a major period of recycling of ancient crustal materials in the NCC. And also indicate that the NCC probably not has developed a long-lived subduction to complete the final assembly of the NCC. The main reason is that the radiogenic Hf composition of detrital zircons of the Paleoproterozoic metasedimenatry from the Trans-North China Orogen marked a progression towards decreasingly juvenile Hf isotope compositions during the period of 2.5–1.8 Ga, but no a progression towards increasingly juvenile Hf isotope compositions towards depleted mantle-like compositions. Correspondingly, as demonstrated by the yellow array of line in Figure 5(b), the detrital zircons record a long-term shift toward decreasingly positive $\varepsilon_{Hf}(t)$ values, reflecting there were no successive juvenile crustal additions from the underlying convecting mantle wedge during ongoing subduction. Alternatively, the reduction of the radiogenic Hf isotope could be attributed to the rollback of the subducting slab. If the ongoing subducting plate rolled back, the subduction angle and speed would obviously changed [68], leading to the general reduction of radiogenic Hf of detrital zircons from metasedimentary rocks in the Trans-North China Orogen.

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Supporting Information

Table S1 LA-ICPMS U-Pb data for detrital zircons from the Tietonggou Formation

Table S2 Lu-Hf isotopic compositions of detrital zircons from the Tietonggou Formation

Table S3 δ^{18}O compositions of dated detrital zircons of Tietonggou Formation

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